

Presented at the John Dorn Memorial
Symposium AIME, Cleveland, Ohio,
October 16-19, 1972

LBL-1174

c.1

THE STRUCTURE AND PROPERTIES OF A NON-CARBON
CONTAINING BCC IRON ALLOY AT ROOM AND
ELEVATED TEMPERATURES

V. F. Zackay, E. R. Parker, and D. Bhandarkar

October 1972

Prepared for the U.S. Atomic Energy Commission
under Contract W-7405-ENG-48

For Reference

Not to be taken from this room



LBL-1174

c.1

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

0 0 0 0 3 8 0 0 2 3 2

THE STRUCTURE AND PROPERTIES OF A NON-CARBON CONTAINING BCC
IRON ALLOY AT ROOM AND ELEVATED TEMPERATURES

V. F. Zackay, E. R. Parker, and D. Bhandarkar

ABSTRACT

A Laves phase, Fe_2Ta , was utilized to obtain good room and elevated temperature properties in an iron based, carbon-free, BCC alloy containing one atomic percent Ta and 7 atomic percent Cr. The Fe_2Ta precipitate was spheroidized to enhance room temperature ductility and toughness; it was stable enough at high temperatures to provide good creep resistance. The alloy had a yield strength of 61,000 psi at room temperature, but the yield strength did not drop precipitously until temperatures in the neighborhood of 1300°F were reached. Stress rupture tests carried out at various constant loads and temperatures indicated that the alloy could withstand reasonably high stresses for long periods of time at temperatures up to about 1100°F. Above about 1100°F the creep strength dropped to low values. A comparison with the mechanical properties of existing materials indicated that the Fe-Ta-Cr alloy had creep properties superior to many steels currently used in the 600°-1100°F temperature range. The superior properties were attributed to the stabilization of the matrix substructure by the Fe_2Ta precipitate which is much more stable in this temperature range than the carbides used for strengthening currently used steels. Several heat treatments were investigated in order to optimize properties. Information gained from research on the Fe-Ta-Cr alloy is being utilized to develop additional alloys. Some of these are expected to be useful up to 1500°F.

THE STRUCTURE AND PROPERTIES OF A NON-CARBON-CONTAINING
BCC IRON ALLOY AT ROOM AND ELEVATED TEMPERATURES

V. F. Zackay, E. R. Parker and D. Bhandarkar

Department of Materials Science and Engineering
College of Engineering; University of California,
Berkeley, California

INTRODUCTION

Most commercial creep resistant materials derive their strength from a fine dispersion of a second phase which hinders the movement of dislocations. In creep resistant ferritic steels, the fine dispersion almost invariably consists of carbides, whereas in high nickel content superalloys, the dispersed phase may be an intermetallic compound such as Ni_3Ti . The particle size, distribution and volume fraction of the second phase, the degree of its coherency, and the mechanical properties of the matrix all affect the creep resistance of dispersion strengthened alloys. The thermal stability of the second phase particles is also an important factor(1).

The morphology and composition of carbide particles tend to change slowly at elevated temperatures and this often results in an increase in creep rate. An example of such an effect is the loss in creep strength of 0.1C-1.0Cr-0.5 Mo steel after long time exposure at service because of the formation of an M_6C type of carbide(2).

The concept of using intermetallic compounds, rather than carbides, for increasing the high temperature strength of alloys is decades old, but progress toward this object has been slow. The status of such work was summarized recently by Decker in his paper on superalloys(3). The

compound $\text{Ni}_3(\text{Al}, \text{Ti})$ plays an important strengthening role, but carbon in amounts ranging from 0.05 to 0.15 percent is considered to be essential for high temperature strength. Experiments in which carbon was reduced to very low levels in superalloys resulted in "...sharply reduced creep life and ductility...."(3).

As previously mentioned, carbide particles coarsen at relatively rapid rates at creep temperatures, but according to Decker so do particles of $\text{Ni}_3(\text{Al}, \text{Ti})$. The effects of other intermetallic compounds such as the sigma phase have also been investigated by Mihalisin et al(4), who found that the presence of sigma lowered both stress-rupture and room temperature strengths. Decker also summarized the status of knowledge about the effects of the Laves phase particles on strength. He concluded that in general, when the Laves phase is present in significant amounts it "...can degrade room temperature ductility with little effect on creep properties." The results from the present investigation show, however, that with microstructural control, satisfactory values of room temperature strength and ductility, as well as good stress-rupture life, can be attained in very low carbon alloys strengthened with the Laves phase Fe_2Ta .

Recently it was demonstrated by Jones, et al(5) that the brittleness which was due to the presence of a continuous network of the Laves phase at the ferrite grain boundaries, could be overcome by a simple heat treatment which spheroidized the precipitate. The objective of the present investigation was to explore means for producing ferritic alloys without deleterious grain boundary networks of the Laves phase.

The transformation temperatures, $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \delta$, are high in binary iron-tantalum alloys, and it was considered desirable to lower these temperatures to a range more suitable for heat treatments by adding a third element to the alloy. Chromium was chosen for this purpose; its effect is shown in Table I(6).

EXPERIMENTAL PROCEDURE

The alloy with a nominal composition of one atomic percent Ta and 7 atomic percent Cr in iron was chosen for creep studies. It was melted in an induction furnace and cast into 20 lb. ingots in an argon atmosphere. The ingots were homogenized at 1100°C for 3 days and forged at 1100°C into plates 9/16" thick. Specimen blanks for creep tests were cut from these plates; they were rolled to 1/4" thick sheets for tensile specimens. The blanks were encapsulated in quartz tubes with an argon atmosphere and solution treated for 1 hour at 1320°C. This was followed by a hot water (70°C) quench and an aging treatment in a salt bath at 700°C for 40 minutes. Stainless steel bags were used to protect the blanks during the subsequent spheroidizing treatment, which was carried out at 1100°C. The complete heat treating cycle is schematically shown in Fig. 1.

Uniaxial tensile tests were conducted on flat specimens 0.2 inch thick and a gage section of one inch. An Instron outfitted with a furnace and an inert atmosphere chamber was used for elevated temperature tensile tests. The strain rate employed was 0.04 per minute. Constant load creep and stress-rupture tests were carried out on cylindrical specimens that had a 1/4" diameter, 1.5 inches long gage section. Strains were measured with a dial gage extensometer.

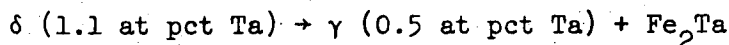
Table I. Phase Transformation Temperatures for Fe-Ta-Cr Alloys

| Alloy Composition | Phase Transformations and Temperatures (°C) | | |
|----------------------------|--|-----------------------------|---|
| | $\alpha \rightarrow \gamma$ | $\gamma \rightarrow \delta$ | $\gamma + \delta \rightarrow \delta$ or $\text{Fe}_2\text{Ta} + \delta \rightarrow \delta$ |
| Fe-1 at pct Ta | 974 | 1238 | 1340 |
| Fe-1 at pct Ta-3 at pct Cr | 890 | 1230 | 1300 |
| Fe-1 at pct Ta-5 at pct Cr | 880 | 1210 | 1280 |
| Fe-1 at pct Ta-7 at pct Cr | 870 | 1160 | 1250 |

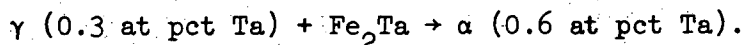
The fracture surfaces of tensile and creep specimens were studied in a JEOL scanning electron microscope. Optical metallography was employed for structural studies, and dilatometry was used for determining transformation temperatures.

RESULTS AND DISCUSSION

The Fe-rich portion of the equilibrium phase diagram of the Fe-Ta system is shown in Fig. 2(7). There are three solid solution regions, α (bcc), γ (fcc) and δ (bcc). The intermetallic compound, Fe_2Ta , is a Laves phase, and it has an hexagonal MgZn_2 structure. The δ phase decomposes eutectoidally at 1293°C in accordance with the following reaction:



The γ phase reacts peritectoidally with Fe_2Ta at 974°C to produce the α solid solution:



Chromium additions change the compositions and temperatures of these transformations, as shown in Table I. The $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \delta$ transformation temperatures were obtained by dilatometry and the $(\gamma + \delta) \rightarrow \delta$ and $(\text{Fe}_2\text{Ta} + \delta) \rightarrow \delta$ temperatures were determined by metallography.

Microstructures of the Fe-1Ta-7Cr alloy are shown in Fig. 3 for various stages of heat treatment. The structure of the alloy in the solution treated condition was characterized by large grains of the retained δ phase, (approximately 1 mm in average grain diameter), and by an almost continuous grain boundary network of precipitate. When the alloy was aged at 700°C , additional precipitate formed within the grains, but there was no noticeable change in the grain boundary network.

Microhardness was measured after various aging times; there was a maximum at 40 minutes, as shown in Fig. 4(6). The microstructure of the alloy in the peak hardness condition is shown in Fig. 3(a). When the aged alloy was heated to 1100°C, the matrix transformed to the face centered cubic solid solution, and the grain boundary precipitate spheroidized. On cooling from the γ phase field, the matrix again transformed to α , with a consequent refinement in grain size. The structure after this treatment consisted of spheroidized precipitate particles in a matrix of α , as shown in Fig. 3(b).

The tensile properties of the Fe-1Ta-7Cr alloy, after aging at 700°C for 40 minutes, are plotted as a function of the test temperature in Fig. 5. Both the yield strength and the ultimate tensile strength became lower as the test temperature was increased. The decrease in yield strength was gradual up to about 600°C; at this temperature the yield strength was about 70 percent of the room temperature value. Both the yield and the ultimate strengths decreased more rapidly as the test temperature was increased above 600°C. The scanning electron fractograph of the tensile specimen tested at 22°C is shown in Fig. 6(a). Fracture occurred in a brittle fashion along the precipitate network at the grain boundaries. More ductile behavior was observed as the test temperature was raised (Fig. 6(b), (c) and (d)).

The room temperature fracture behavior was changed from brittle to ductile by the spheroidizing heat treatment that followed aging. The fracture surface of the tensile specimen tested at room temperature after spheroidizing at 1100°C for 10 minutes is shown in Fig. 7. In

this figure, there is evidence of a significant amount of plastic deformation, although the size of the dimples visible in Fig. 7 did not correspond to the size of the large grains of the δ phase that existed before spheroidizing. Voids apparently were nucleated at the interfaces between Fe_2Ta particles and the matrix, and the microcracks thus formed presumably controlled the dimple size. The yield strength, tensile strength, and elongation are plotted as a function of the test temperature in Fig. 8 for the alloy in spheroidized condition. The results were similar to those shown in Fig. 5 for the aged alloy except that the values of all properties were higher. The improvements were due to the combined effects of spheroidization of the grain boundary precipitate and the reduction in grain size.

Short time tensile properties do not provide adequate information about high temperature strength, and so creep-rupture tests were made on the Fe-1Ta-7Cr alloy. The temperatures selected for evaluation were 1000 and 1100°F (538 and 593°C). Stress vs time to rupture data are plotted in Figs. 9 and 10. Also shown in the figures are the stress-rupture properties reported in the literature for 1 Cr-1Mo-0.25V steel and 403, 410 and 422 stainless steels(8). Both at 1000°F and 1100°F, the Fe-1Ta-7Cr alloy results were above those reported for the 403 and 410 stainless steels, but below those for the 422 stainless steel and the 1 Cr-1Mo-0.25V steel.

The strengthening effect of molybdenum in solid solution in ferrite is well known, so 0.5 percent of molybdenum was added to the Fe-1Ta-7Cr basic composition in another alloy. The expected strengthening was

found when stress-rupture tests were made at 1100°F on the modified alloy. The results are shown in Fig. 10(9).

The preliminary results obtained to date with Laves-phase strengthened alpha iron have clearly shown that such alloys can be made ductile at both room and elevated temperatures. Also, there is an indication from the results of the 0.5 percent molybdenum alloy that with optimization of creep and stress-rupture properties by alloy modification, Laves-phase alloys may find a useful place among high temperature engineering materials.

ACKNOWLEDGMENTS

The authors wish to express their deep appreciation to Professor R. M. Fulrath for his help and guidance in the scanning electron microscope studies. We would also like to thank S. Jin and M. S. Bhat for permission to use previously unpublished data.

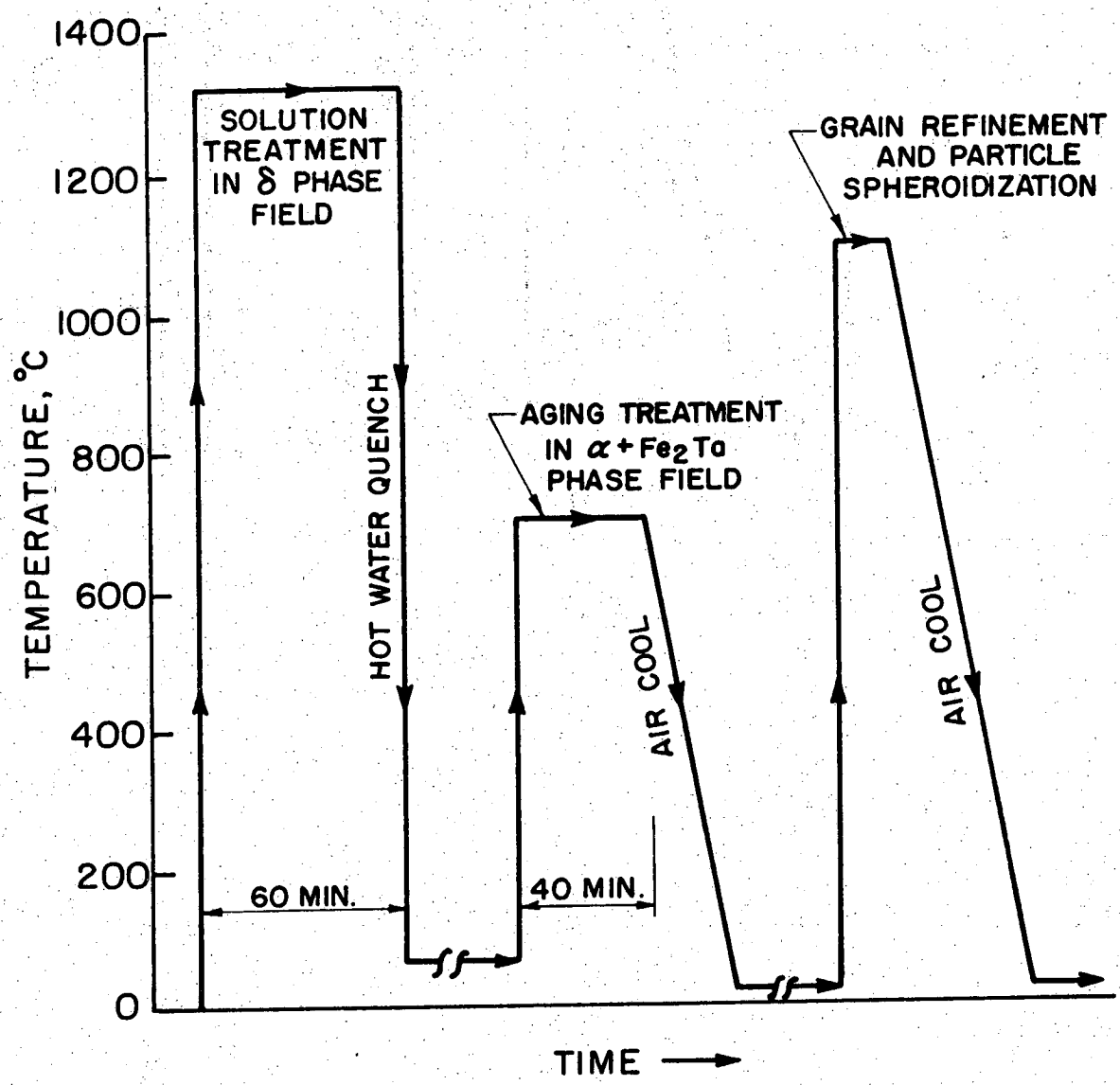
This work was performed under the auspices of the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory at the University of California, Berkeley, California.

REFERENCES

1. R. W. Guard: Mechanical Behavior of Materials at Elevated Temperature, McGraw-Hill Book Co., Inc., 1961, ed. J. E. Dorn, p. 270.
2. L. H. Toft and R. A. Marsden: Iron Steel Inst. Spec. Report No. 70, 1961, p. 276.
3. R. F. Decker: Steel-Strengthening Mechanisms, Symp. sponsored by Climax Molybdenum Co., 1969, p. 147.
4. J. R. Mihalisin, C. G. Bieber and R. T. Grant: Trans. AIME, 1968, vol. 242, p. 2399.
5. R. H. Jones, E. R. Parker and V. F. Zackay: Proc. Fifth Intern. Mater. Symp., Berkeley, Calif., September 1971, p. 829.
6. S. Jin: M.S. Thesis, LBL Report 443, University of California, Berkeley, December 1971.
7. A. K. Sinha and W. Hume-Rothery: J. Iron Steel Inst., 1967, vol. 205, p. 671.
8. Metals Handbook, vol. 1, 8th ed., Amer. Soc. for Metals, Metals Park, Ohio, 1969.
9. M. S. Bhat: unpublished research.

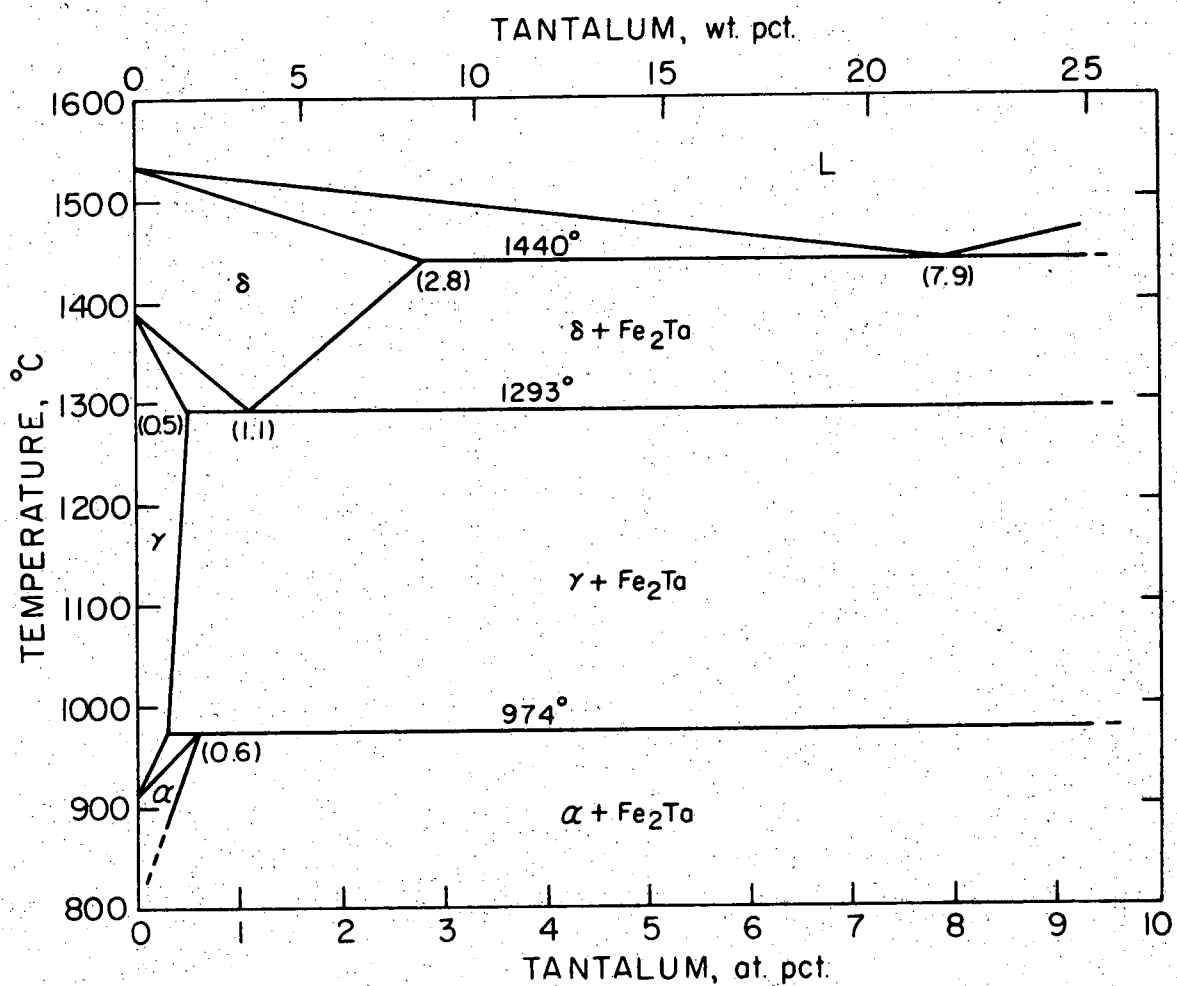
FIGURE CAPTIONS

1. Heat treating cycle for the Fe-1Ta-7Cr alloy.
2. Iron-rich portion of the equilibrium phase diagram of the Fe-Ta system.
3. Microstructures of the Fe-1Ta-7Cr alloy (a) Aged at 700°C for 40 minutes (b) Aged at 700°C and spheroidized at 1100°C for 10 minutes.
4. Hardness vs aging time curve for the Fe-1Ta-7Cr alloy aged at 700°C.
5. Yield strength, ultimate tensile strength and elongation to fracture of the Fe-1Ta-7Cr alloy, aged at 700°C for 40 minutes, at the indicated test temperatures.
6. Scanning electron fractographs of the Fe-1Ta-7Cr alloy, aged at 700°C for 40 minutes, tested at temperatures (a) 22°C (b) 200°C (c) 400°C, and (d) 600°C.
7. Scanning electron fractograph of the Fe-1Ta-7Cr alloy, spheroidized at 1100°C for 10 minutes, tested at 22°C.
8. Yield strength, ultimate tensile strength and elongation to fracture of the Fe-1Ta-7Cr alloy, spheroidized at 1100°C for 10 minutes, at the indicated test temperatures.
9. Stress vs rupture time at 1000°F (538°C) for the spheroidized Fe-1Ta-7Cr alloy. Also shown are the results reported in the literature for the 1Cr-1Mo-0.25V steel and the 403, 410 and 422 stainless steels(8).
10. Stress vs rupture time at 1100°F (593°C) for the alloys mentioned in Fig. 9. Also shown are the preliminary results on the Fe-1Ta-7Cr-0.5Mo alloy.



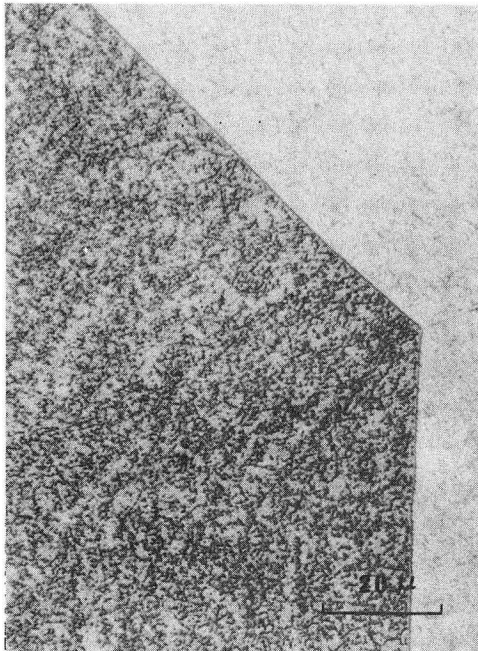
XBL 7210-7049

Fig. 1

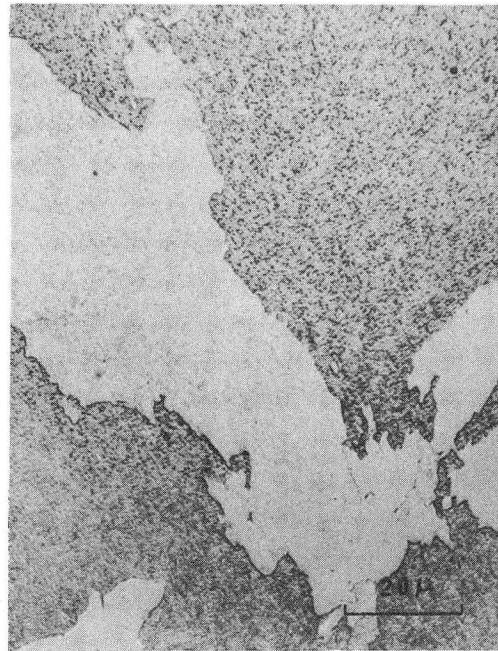


XBL 721-5996

Fig. 2

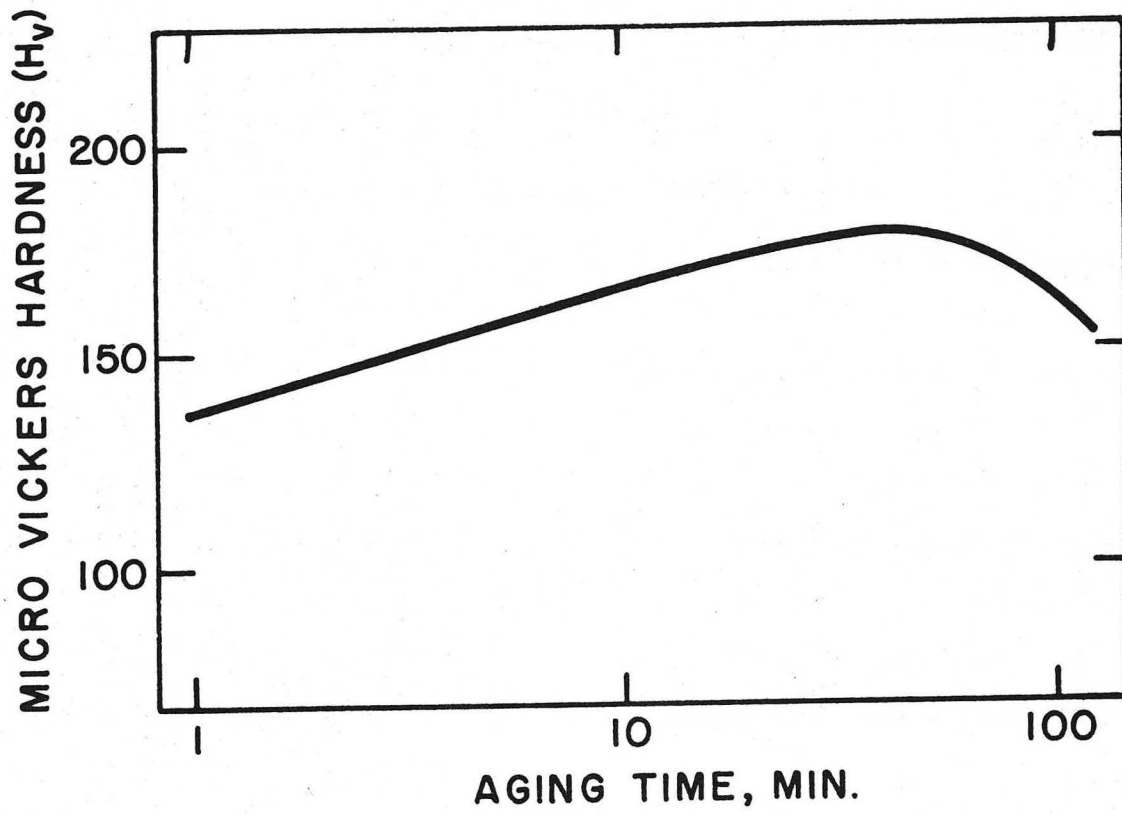


(a)



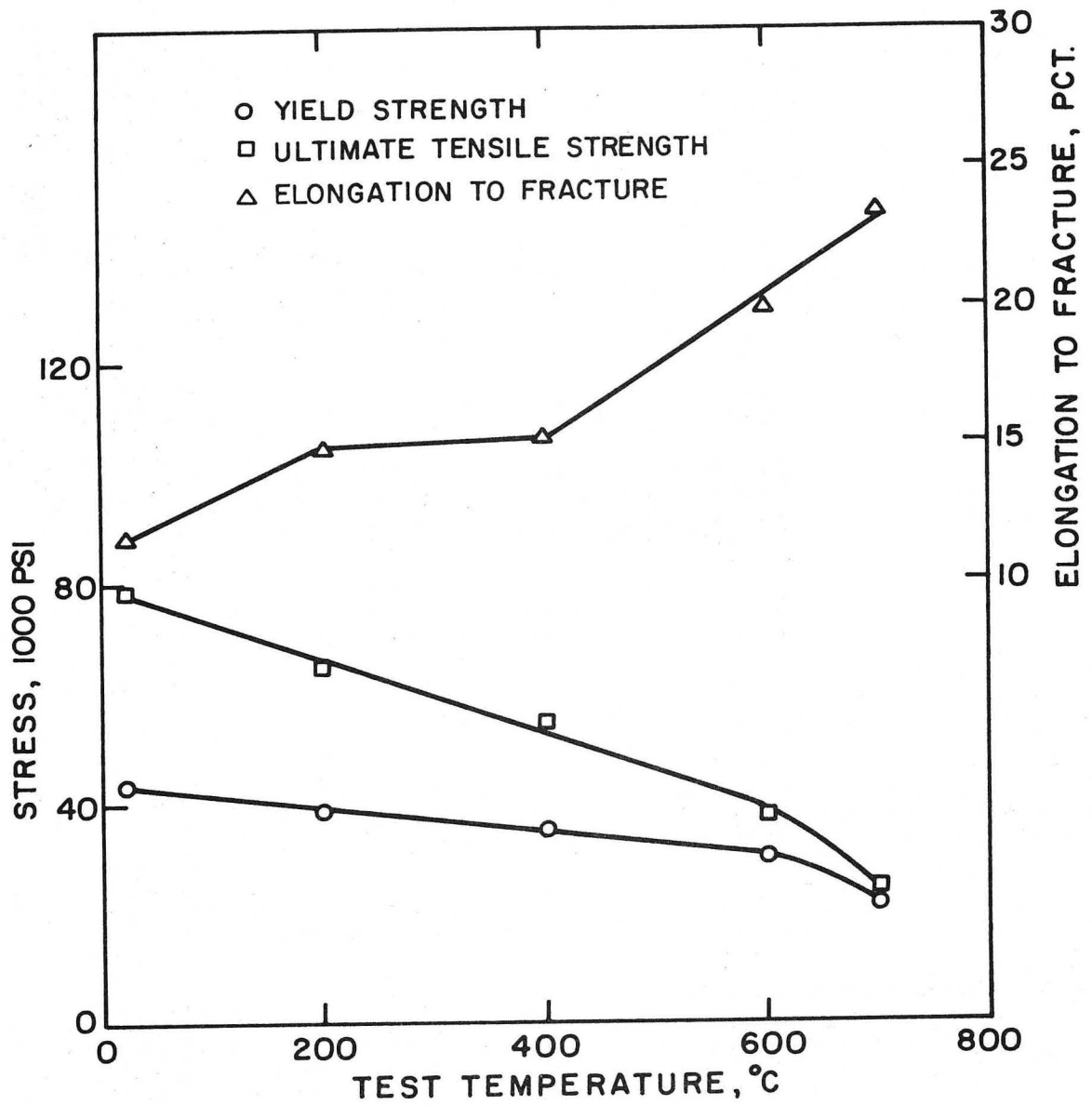
XBB 7210-4984
(b)

Fig. 3



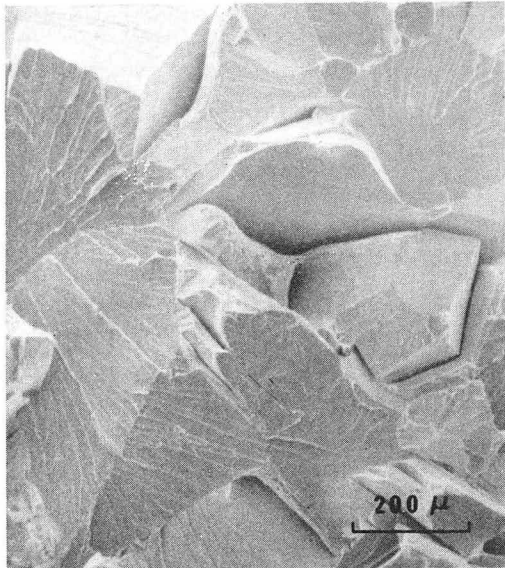
XBL 7210-7050

Fig. 4

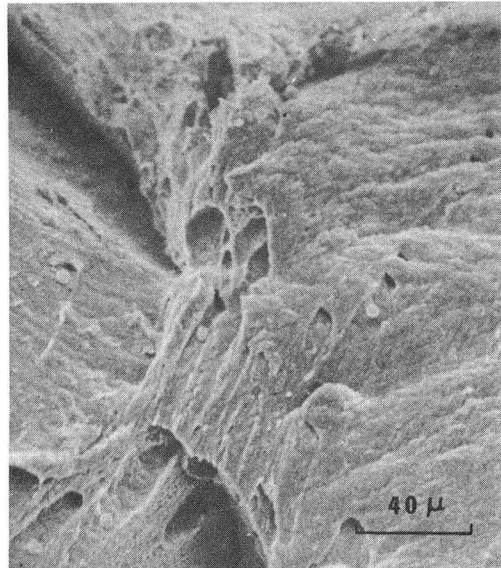


XBL 7210-7051

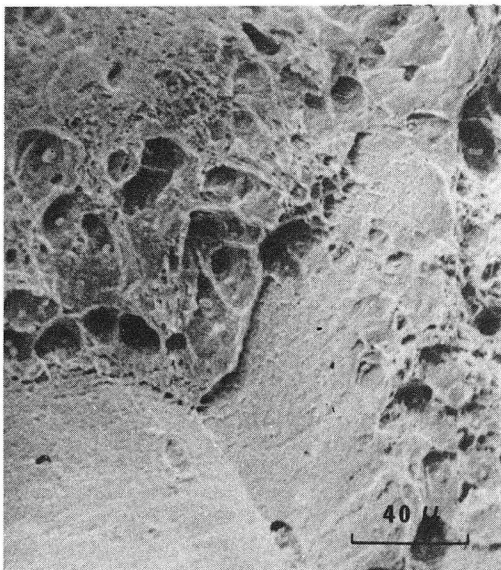
Fig. 5



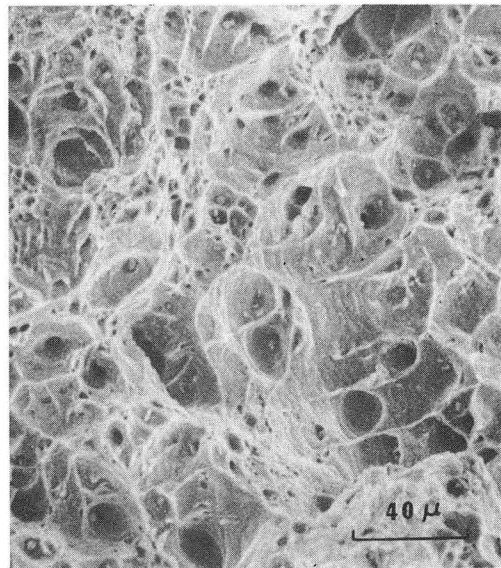
(a)



(b)



(c)

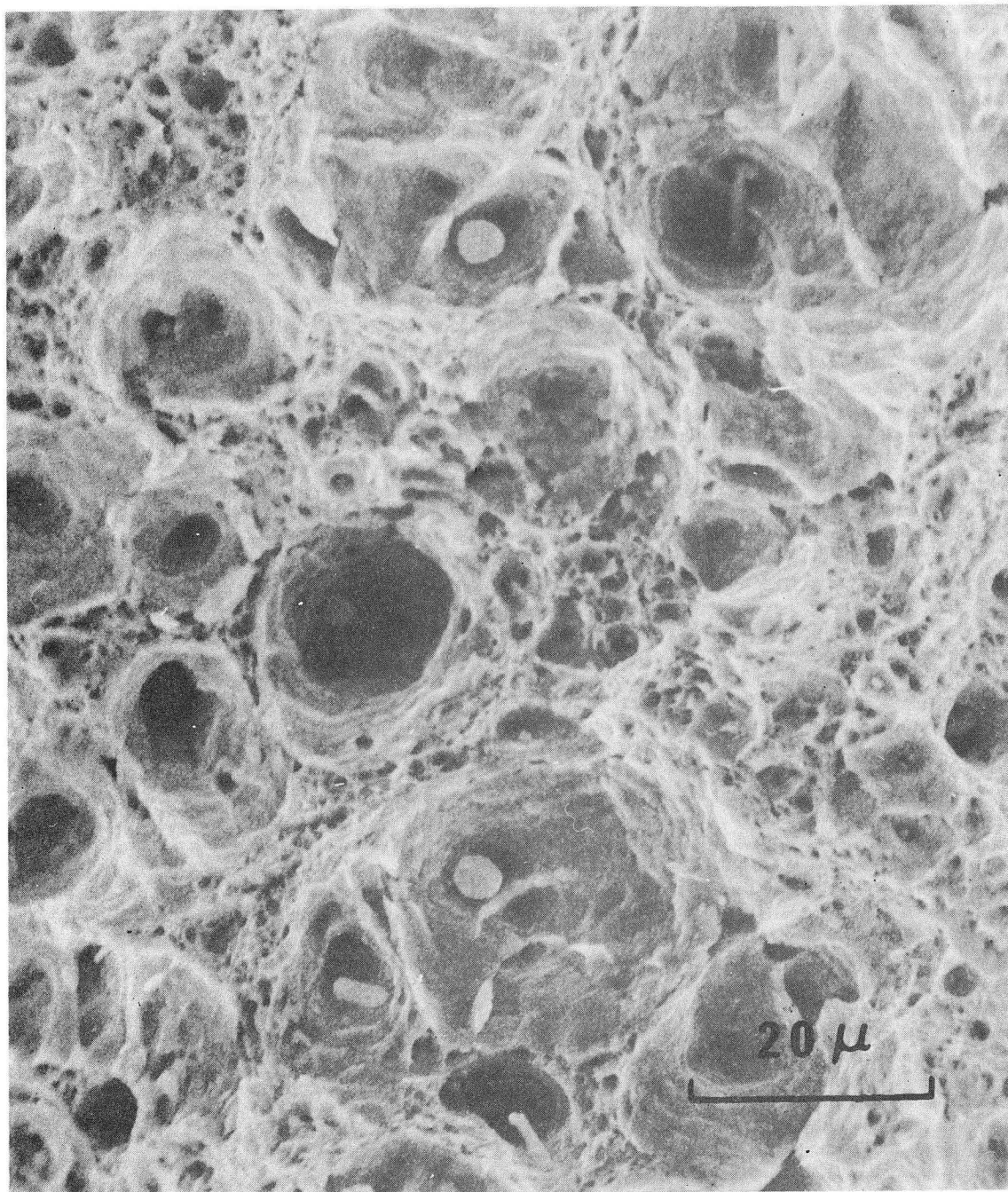


(d)

XBB 7210-4992

Fig. 6

-17-



XBB 7210-4991

Fig. 7

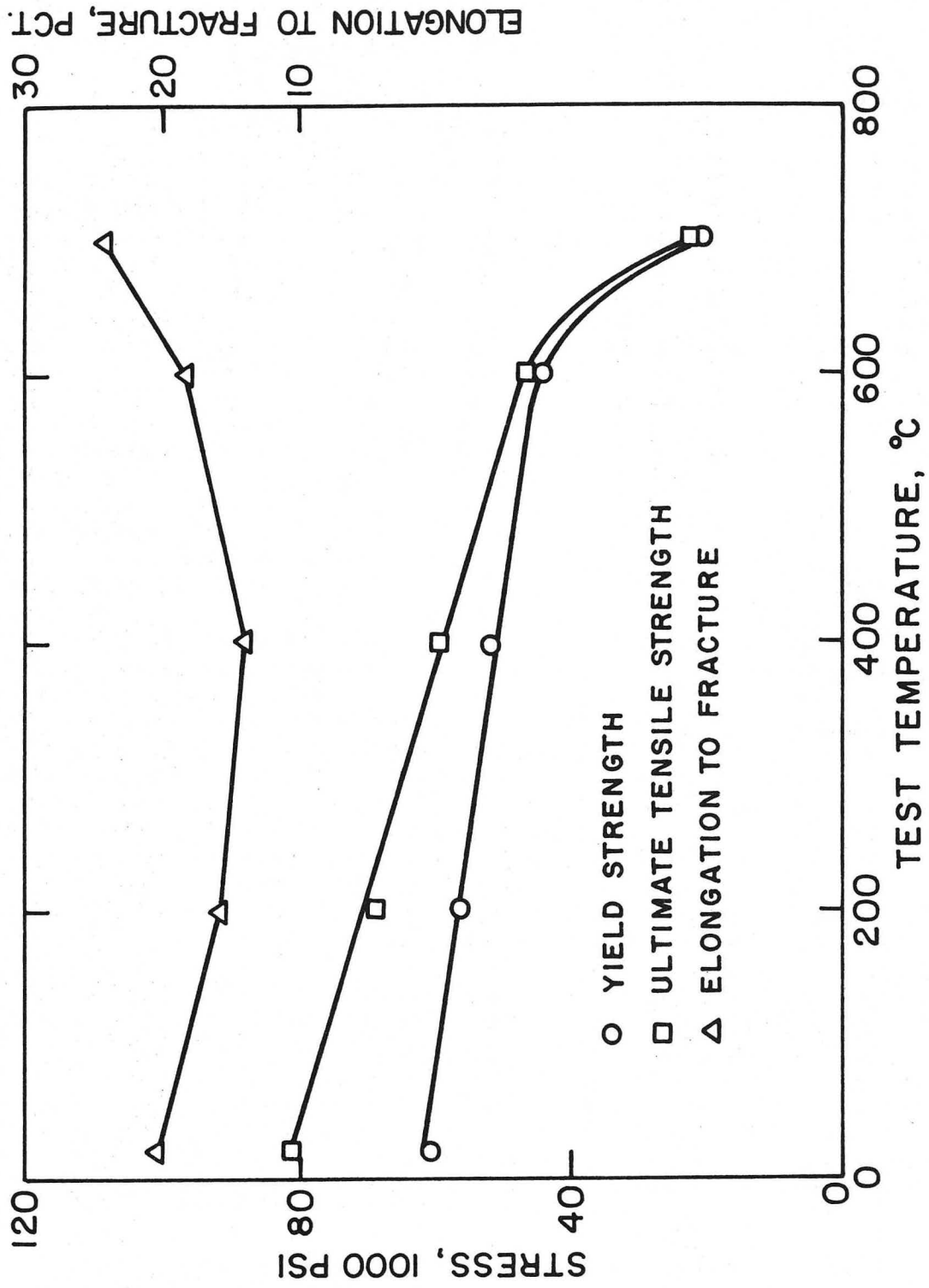
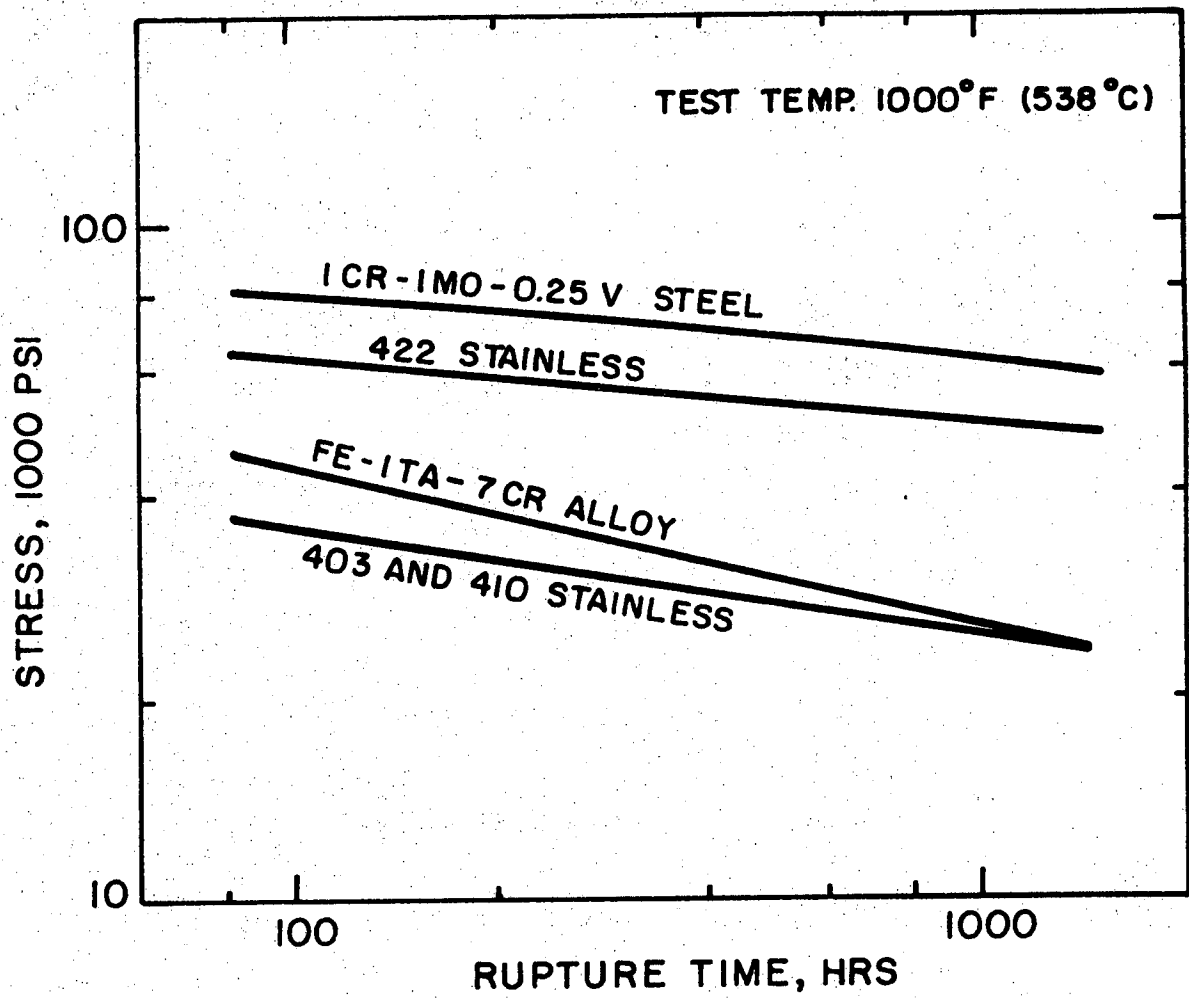


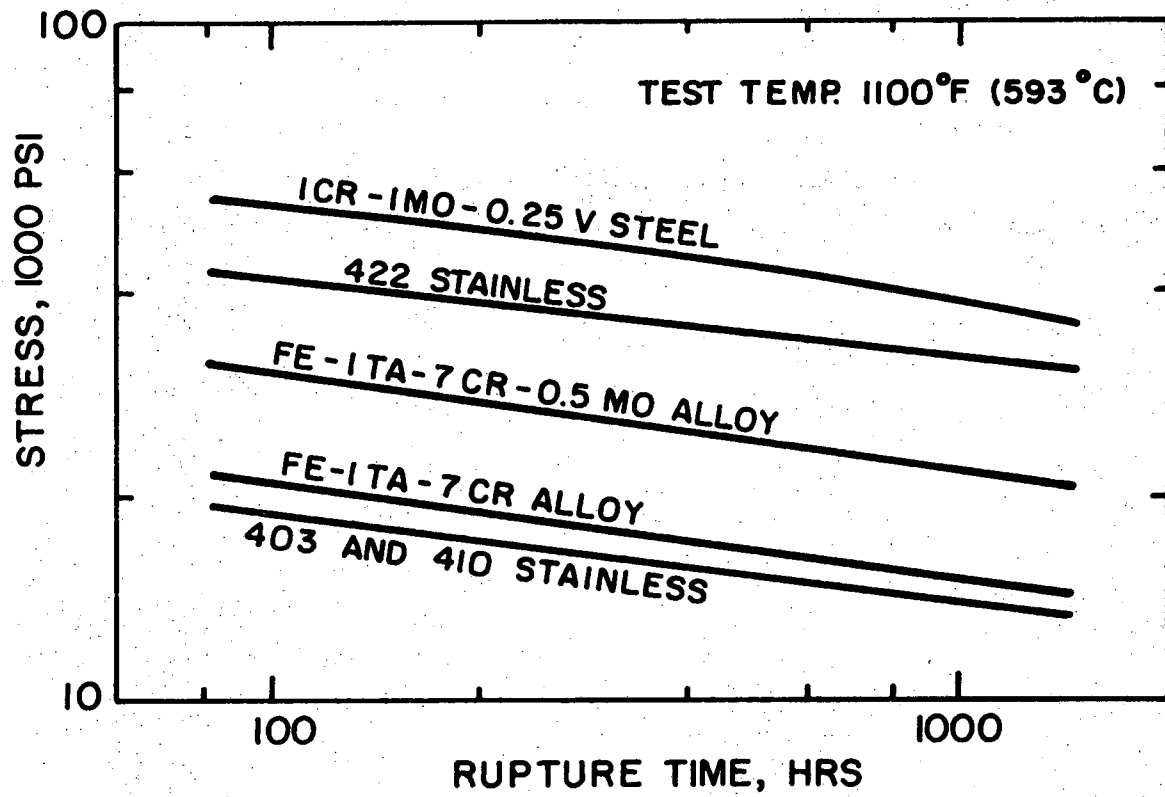
Fig. 8

XBL 7210-7052



XBL 7210-7053

Fig. 9



XBL 7210-7054

Fig. 10

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

TECHNICAL INFORMATION DIVISION
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720